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Short Communication

Rapid preparation of TiC reinforced Ti6Al4V based composites by carburizing method through spark plasma sintering technique

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ABSTRACT

A rapid method, using woven carbon fiber cloth as carbon supplier, combining with spark plasma sintering (SPS) technique, was proposed to fabricate TiC reinforced Ti6Ai4V based composites. By this way, carburized layer with the thickness of 4 mm can be obtained within 20 min. Microstructure analysis reveals that the composites exhibit advantage of good interfaces between the uniformly distributed TiC precipitates and matrix. Investigations on mechanical properties show that compared with Ti6Al4V matrix alloy, the TiC reinforced Ti6Al4V based composites exhibits higher yield strength, higher ultimate compressive strength, and similar ductility under both quasi-static compression and dynamic compression. The failure mechanisms of the composites and its influence on mechanical properties were discussed.

1. Introduction

Due to the high specific strength, excellent fatigue properties, good corrosion resistance and acceptable fracture toughness, titanium alloy has been widely used in biomedical and aerospace industry as well as in the chemical and automobile industry. However, poor tribological properties such as high friction coefficient and low hardness limit the extensive application of Ti–6Al–4V alloy in these engineering applications [1–3]. Preparing titanium alloy matrix composites reinforced with ceramics particles has been proved to be an effective way to improve the mechanical properties. Among various ceramic particulates, TiC is expected to be an excellent reinforcement for Ti6Al4V based composites due to its good properties, such as high chemical stability, high thermal stability, good corrosion resistance and high hardness [4–6]. A large amount of work [7,8] has been focused on TiC reinforced Ti6Al4V based composites (TiC/Ti6Al4V), and these work has got a remarkable improvement in hardness and tribological properties. However, among the traditional fabricating techniques of TiC/Ti6Al4V composites such as ingot metallurgy and powder metallurgy, the problems including severe interfacial reaction and poor wettability between the reinforcements and matrix are inevitable, which leads to bad ductility of TiC/Ti6Al4V composites.

Carburizing method is supposed to be an effective way to prepare TiC/Ti6Al4V composites since the TiC reinforcements fabricated by carburizing method can distribute uniformly in matrix and combine well with matrix [2,9]. However, the thickness of formed carburized layer is very thin. Wu et al. [10] investigated the method of electrolytic molten salt carburization and obtained a carburized layer with the thickness of 70 μm. Kima et al. [3] obtained a carburized layer which increase to 150 μm by plasma carburizing process. Methane and acetylene are commonly used as the carbon supplier, and hydrogen brittleness is an inevitable consequence derived from the utilizations of the traditional carbon supplier [2]. Moreover, carburizing treatment is a time consuming process. So far, the conventional carburizing methods are inefficiency, and it is difficult to obtain a bulk TiC/Ti6Al4V composites by carburizing method. If there is an effective way that can enable the carbon atoms diffuse into the matrix deeply at a rapid rate, the efficiency of preparing TiC/Ti6Al4V composites will be enhanced remarkably.

In the present study, a very rapid method is proposed to fabricate TiC reinforced Ti6Al4V based composites. In this method, SPS technique was employed, and woven carbon fiber cloth was selected as carbon supplier. Advantages of applying the SPS technique include its moderate uniaxial pressure and rapid sintering, besides, pulse current may be beneficial for the diffusion of carbon atoms [11,12]. The reason of selecting woven carbon fiber cloth as carbon supplier is that C–C chemical bonding energy within carbon fibers (bond energy < 200 kJ/mol) are weaker than that of the hexagonal graphitic (bond energy > 500 kJ/mol) [13], and the weaker

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bonds of C–C within carbon fiber are easily broken up at high temperature, which is beneficial for supplying adequate carbon atoms during the carburizing process. After TiC/Ti6Al4V composites were prepared by SPS method, mechanical properties including Brinell hardness and compressive strength were measured to reveal the effect of TiC reinforcement on the mechanical properties of TiC/Ti6Al4V composites. Failure mechanism of the composites after dynamic compression is also discussed.

2. Experiments

Ti6Al4V(Ti alloy with 6 at.%Al and 4 at.%V) sample with a dimension of Φ30 mm × 4 mm was sliced from round bar stock by electrical discharge wire cutting machine. Woven carbon fiber cloths were cut into round slices with diameter of 30 mm. The cylindrical Ti6Al4V specimen together with two pieces of carbon fiber cloth covered on both sides were put into a cylindrical graphite die. The specimens were fabricated at 1500 °C with a heating rate of 100 °C/min and a maintained uniaxial pressure of 50 MPa. To further explore the effects of holding time on the microstructure and mechanical properties of TiC/Ti6Al4V composites, two kinds of specimens were fabricated with different holding times of 10 min and 20 min, respectively.

Brinell hardness were conducted using a 2.5 mm-diameter steal ball at a load of 187.5 kgf (1.839 kN) applied for a period of 30 s. Uniaxial quasi-static compressive experiments were conducted at strain rate of 10^{-3} s^{-1}. Uniaxial dynamic compressive experiments were conducted using Split Hopkinson Pressure Bar at an average strain rate of 3500 s^{-1}. The experiment device and methods have been introduced in detail in literature [14,15]. The retrieved specimens after dynamic compression were cut along the loading direction. Scanning electron microscopy (SEM) was employed to characterize the microstructure of the prepared TiC/Ti6Al4V composites and the retrieved specimens after dynamic compression. The X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used to analyze the phases and interfaces of prepared composites.

3. Results and discussion

3.1. Microstructure characterization

Fig. 1 shows the SEM micrographs of the TiC/Ti6Al4V composites fabricated at 1500 °C with the pressure of 50 MPa. Fig. 1a displays the microstructure of the TiC/Ti6Al4V composites fabricated at 1500 °C for 10 min, it can be observed that the strip precipitates distribute in Ti6Al4V matrix homogeneously, and the length of the precipitates ranges from 10 μm to 100 μm with a width of about 2 μm. Fig. 1b displays the microstructure of the TiC/Ti6Al4V composites fabricated at 1500 °C for 20 min. By comparing Fig. 1b with Fig. 1a, it can be seen that with the holding time increased from 10 min to 20 min, the morphology and distribution of precipitates shows no obviously change. However, the quantity of the precipitate is remarkably increased. Fig. 1c shows enlarged SEM micrograph of precipitate in matrix. It can be observed that the interfaces between the precipitates and matrix are in good adhesion without any cracks or pores, indicating a good interface bonding. Another important observation should be pointed out, the precipitates displayed in Fig. 1 shows the shape of curved strips, thus it can be deduced that the precipitates have the shape of curved sheets in three dimensional spaces.

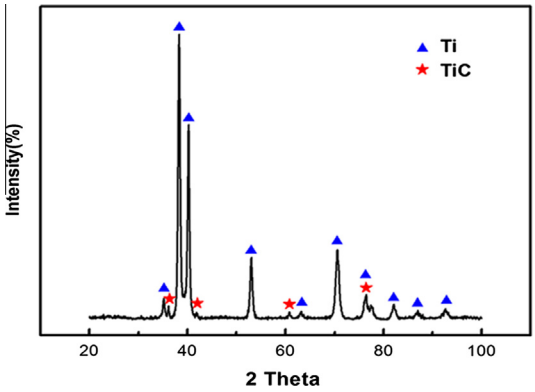

Fig. 2. XRD results of prepared materials.

Fig. 2 shows the XRD results of the TiC/Ti6Al4V composites. The current composites mainly consist of α-Ti phase and TiC phase, and no other new phases are detected, indicating that the observed precipitates (shown in Fig. 1) may be TiC.

In order to identify the phase structure of the precipitates, TEM was employed. Fig. 3a shows a TEM micrograph of prepared TiC/Ti6Al4V composites. Fig. 3b and c shows the selected area electron diffraction (SAED) patterns taken from the circular areas in Fig. 3a. In Fig. 3b, SAED patterns taken from area A indicate that the matrix is α-Ti. From Fig. 3c (SAED patterns taken from area B which contains both matrix and precipitate), it can be found that both the SAED patterns of α-Ti and TiC were obtained. Therefore, it can be finally confirmed that the strip-like phase is TiC. Moreover, from Fig. 3a, clean interface without any unfavorable reaction and interfacial cracking between precipitates and matrix was observed, indicating a good interface bonding.

Based on above analysis, TiC/Ti6Al4V composites with good interface bonding between TiC precipitates and matrix were successfully prepared by carburizing method. Moreover, the holding time is an important parameter that influences the microstructure of the composites. It is reasonable to predict that with the increase of holding time, the carbon atoms will diffuse into the matrix much more deeply, and the quantity of the precipitate will increase remarkably.

3.2. Mechanical properties

Fig. 4 displays the hardness test results of TiC/Ti6Al4V composites. For comparison, Ti6Al4V alloy was also tested. It can be clearly

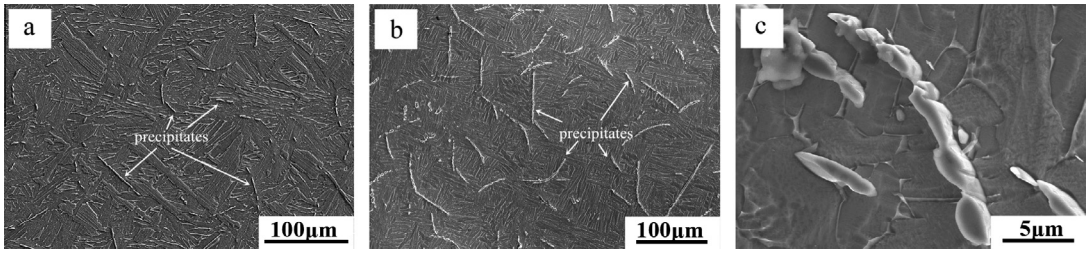

Fig. 1. SEM micrographs of specimens fabricated at 1500 °C for: (a) 10 min, and (b and c) 20 min; (c) is enlarged SEM micrograph showing details of precipitates.

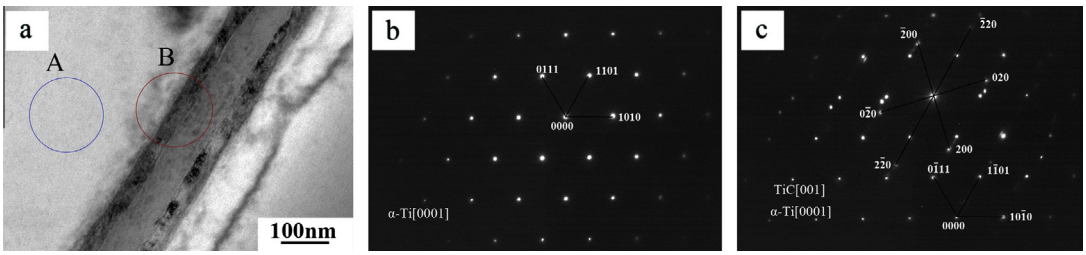

Fig. 3. (a) TEM micrograph of prepared materials; (b) SAED pattern taken from area A in a; (c) SAED pattern taken from area B in a.

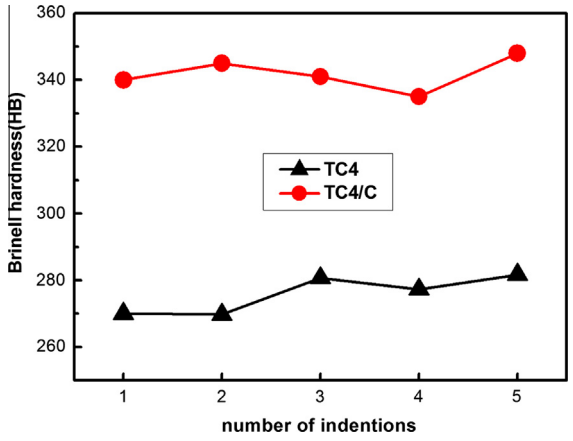

Fig. 4. Brinell hardness of Ti6Al4V matrix alloy and TiC/Ti6Al4V composites.

seen that the hardness values of TiC/Ti6Al4V composites maintain at about 345HB which is nearly 70HB higher than that of the matrix alloy. According to Rastegari et al. [16], the enhancement of hardness can be attributed to the generation of high volume fraction of TiC reinforcements (precipitation hardening effect) and the increase of carbon content (solution hardening effect).

Fig. 5 shows the true stress–strain curves of TiC/Ti6Al4V composites under both quasi-static compression and dynamic compression. For comparison, Ti6Al4V alloy was also tested. At the strain rate of $10^{-3}\,s^{-1}$ (Fig. 5a), it can be seen that TiC/Ti6Al4V composites show a high yield strength of 1150 MPa, which is higher than 980 MPa of Ti6Al4V matrix alloy, and the ultimate compressive strength of composites is 1490 MPa, which is also higher than 1300 MPa of Ti6Al4V matrix alloy. When subjected to strain rate of $3500\,s^{-1}$ (Fig. 5b), TiC/Ti6Al4V composites show a high yield strength of 1520 MPa, which is much higher than 1330 MPa of Ti6Al4V matrix alloy, and the ultimate compressive strength of composites is 1530 MPa, which is also higher than 1410 MPa of Ti6Al4V matrix alloy. These results are in agreement with the results obtained by Wang Xiang [7] who investigated the properties of TiC/Ti6Al4V composites fabricated by melting-casting process. It was found that the TiC/Ti6Al4V composites show higher compressive strength than that of Ti6Al4V matrix alloy. Huang et al. [17] also reported improved compressive yield strength for the TiC/Ti6Al4V composites compared to Ti6Al4V matrix alloy. Moreover, the failure strain of TiC/Ti6Al4V composites is similar to that of Ti6Al4V matrix alloy under both quasi-static compression and dynamic compression. Thus, the generation of TiC phase can enhance the strength of TiC/Ti6Al4V composites remarkably and keep good ductility.

In order to analysis the failure mechanisms of the TiC/Ti6Al4V composites, the fractured specimens after uniaxial dynamic compression were sectioned along the compression axis, and the

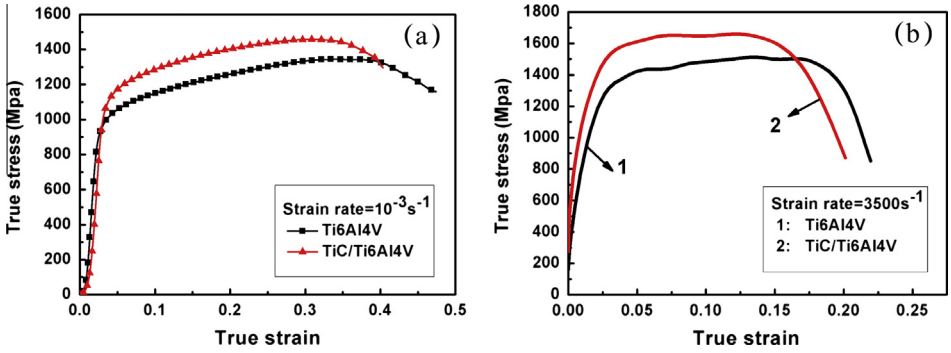

Fig. 5. True stress–strain curves of Ti6Al4V matrix alloy and TiC/Ti6Al4V composites: (a) under quasi-static compression; (b) under dynamic compression.

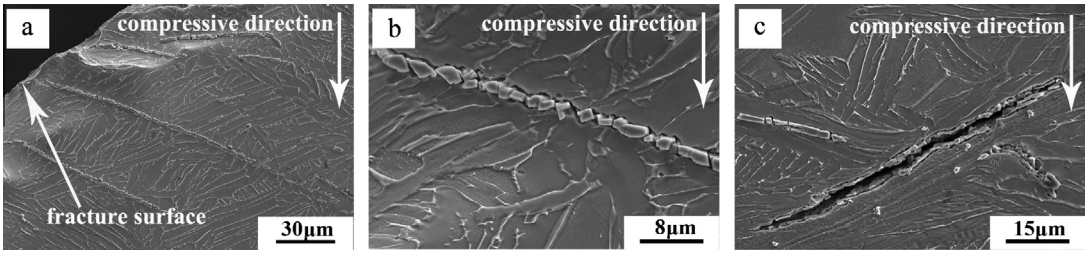

Fig. 6. SEM micrographs of specimens subjected to dynamic compression: (a) showing the fracture surface, (b) and (c) showing micro-crack within TiC.

sectioned surfaces were polished to mirror finish, followed by SEM analyzing. Fig. 6 shows SEM micrographs of specimens after dynamic compression. In Fig. 6a, it is obvious that the failure occurred on the plane of maximum shear stress, indicating that the failure mode is shear failure. An enlarged micrograph in Fig. 6b shows micro-cracks within TiC phase. Fig. 6c shows that the micro-cracks get through the entire TiC precipitate. The results of the present paper are in agreement with that of Antonio et al. [18] in which many cracks were formed within TiC particles during the tensile test. Based on the above observation, the failure mechanism of the present TiC/Ti6Al4V composites can be concluded as below: The micro-cracks of the composite are generated from the hard TiC precipitates. Then these micro-cracks propagate within TiC phase, and extend to the matrix. With the increasing strain, the cracks propagate rapidly under shear stress, leading to a shear failure of composite under dynamic compression.

In a word, the presence of TiC precipitates lead to an increase in strength. Strengthening mechanisms of TiC precipitates are attributed to such factors as load transfer [19], grain refinement [20], solid solution strengthening of carbon in α-Ti phase [7] and geometrically increased density of dislocations around the precipitates [21]. These factors, independently or concurrently, are considered responsible for enhanced the strength of the composites. For the current composites, the observation of failure specimens indicates that during the compressive test, the cracks initiate and propagate within hard TiC precipitates rather than the relatively ductile Ti6Al4V alloy, and the strengthening mechanism of the composites may be mainly attributed to the load transfer from the soft matrix onto the hard precipitate. Therefore, due to the high hardness of TiC, good interface bonding (shown in Fig. 1) and the transfer of stress from the matrix to TiC precipitates, TiC/Ti6Al4V composites exhibit higher yield strength and ultimate compressive strength than those of the matrix alloy.

4. Conclusions

In the present research, a rapid method is proposed to fabricate TiC/Ti6Al4V composites. In this method, SPS technique was employed, and woven carbon fiber cloth was selected as carbon supplier. Due to the good diffusion ability of carbon atoms from carbon fiber and high temperature, moderate uniaxial pressure and the special effect of current pulse provided by SPS technique, carburized layer of Ti6Al4V alloy with thickness of 4 mm can be obtained within 20 min. Microstructure characteristics, mechanical properties and failure mechanisms of the composite are investigated. Results of microstructure observation and mechanical properties tests can be concluded as follows.

(1) Microstructure analysis reveals that TiC participates with shape of curved sheets are distributed uniformly in matrix, and the TiC/Ti6Al4V composites exhibit advantage of good interface bonding between reinforcements and matrix alloy.
(2) Due to second phase strengthening effect, the TiC/Ti6Al4V composites exhibit higher hardness, higher yield strength and higher ultimate compressive strength compared with Ti6Al4V matrix alloy.
(3) The failure mode of TiC/Ti6Al4V composites is shear failure. It can be concluded as below: Micro-cracks generated from TiC precipitates. Then the micro-cracks extend to the matrix under shear stress, and finally lead to a shear failure of composite.

Acknowledgements

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